USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 1225
SOIL SAMPLING FOR VOLATILE ORGANIC COMPOUNDS (VOCs)

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Reviewed by: _____ 2/5/2016
Peter Husby — Date
Biology Team Leader/Technical Director

Reviewed by: _____ 2/5/2016
Lucrina Jones, Laboratory QA Officer — Date

Approved by: _____ 2/8/2016
Duane James, Acting Laboratory Director — Date

Periodic Review:

Signature	Title	Date

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1 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes field collection, handling, and preparation of soil samples for analyses of volatile organic compounds (VOCs) in solid material (e.g., soils, sediments and solid waste). This SOP includes guidance published by EPA Office of Solid Waste (1998) and complements EPA Laboratory Methods 5035 (purge and trap) and EPA 8015, 8021, 8260 (gas chromatography). Specialized equipment and containers are designed to maintain sample integrity for soil or solid materials which may contain contaminants with boiling points less than 200□C.

Research shows that traditional sample collection techniques may result in substantial losses of volatiles. These losses (an order of magnitude or more) are the culmination of volatilization and biodegradation that occur during the sampling, storage, and subsequent sub-sampling in the laboratory. To address significant problems with soil VOC analyses, Method 5035 as revised in Update III of SW-846 (June 13, 1997 Federal Register) and further amended under Method 5035A, July 2002. Samples are handled differently from the onset of sample collection, depending upon the action levels for the project and the anticipated concentrations of VOCs at the site. Field personnel may need to collect samples with both low (<200 ug/kg) and medium (>200 ug/kg) level VOC concentrations. Field preservation and laboratory preservation options are described in order to address sampling for soils at various concentrations.

2 METHOD SUMMARY

Collect approximately 5-25 grams from freshly exposed soil into a specially-designed hand-held coring device, or a pre-weighed/pre-preserved vial and ship to the laboratory for overnight delivery for further preservation and/or analysis. In addition to the samples collected for VOC analysis, collect an additional co-located sample for moisture content determination for results on a dry-weight basis. A brief summary of each method option is presented below.

2.1 Thermal Preservation Low Level (<200 ug/kg)

This method requires either a specially designed coring devise tested to withstand freezing temperatures (i.e., Encore ™) and or further chemical/thermal preservation upon receipt at the laboratory. However, no field chemicals are required. Thermal (freezing) or chemical ($NaHSO_4$) preservation within 24 hours of collection at the laboratory may extend the analysis holding times to two weeks. Detection limits are based on the analyte, method, and laboratory capability, but typically range from 0.5 to 5 ug/kg. Bias may exist for some soil types (e.g., high clay or organic carbon). Additional volume is often required for laboratory QC and percent moisture determinations. Further dilution/analysis requires a sample collected under the medium level methanol preservation procedure summarized below.

2.2 Chemical Preservation Sodium bisulfate ($NaHSO_4$) – Low Level (<200 ug/kg)

This method requires that $NaHSO_4$ solution be added to a pre-weighed and tared vial prior to sample collection. This method is limited to consolidated soils which can be collected in a coring device and cannot be used on carbonaceous soils. The sample container (glass vial) also serves as the purge vessel. Detection limits are based on the analyte, method, and laboratory capability, but typically range from 0.5 to 5 ug/kg. Since the vial is also used as sparge vessel, samples cannot be diluted, and VOA vials cannot be reanalyzed. Bias may exist for some soil types (e.g., high clay or organic carbon). Further dilution/analysis requires a sample collected under the methanol preservation procedure below. Additional volume is often required for laboratory QC and percent moisture determinations. Shipments must comply with special DOT labeling, limited quantities and special packaging requirements.

2.3 Methanol Preservation Medium Level (>200 ug/kg)

This method requires that purge and trap grade methanol be added to a pre-weighed and tared vial prior to sample collection. This method is often referred to as preservation/extraction procedure and holding time is two week from date and time of collection. This method may be used for aggregate and cemented materials by increasing sample size and volume of methanol. Detection limits are based on the analyte, method, and laboratory capability, but typically range from 5 to 50 ug/kg. Additional volume is often required for laboratory QC and percent moisture determinations. Shipments must comply with special DOT labeling, limited quantities and special packaging requirements.

3 DEFINITIONS

There are no terms or definitions specific to this procedure. For terms and acronyms in general use at the EPA Region 9 Laboratory refer to Appendix A of the Laboratory Quality Assurance Plan.

4 RESPONSIBILITIES

Field Team Leader – It is the responsibility of the Field Team Leader to assure that all members of the field team have the proper training and are aware of and follow the guidelines outlined in this procedure and in other applicable documents.

Field Team Members – It is the responsibility of each Field Team Member to be aware of the requirements and follow the guidelines outlined in this procedure.

5 SAFETY & HEALTH

When working with potentially hazardous materials, follow U.S. EPA, OSHA and specific health and safety procedures. More specifically, depending upon the site-specific contaminants, various protective programs must be implemented to ensure the samplers

safety prior to collecting the first sample. The EPA Region 9 Field and Biology (FAB) Team member collecting the sample should not climb into trenches where bank failure may cause him or her to lose their balance. To prevent this, the person performing the sampling should be completed via augers with extensions or from directly from the backhoe immediately after removal from the ambient ground area.

5.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation should be performed in a well-vented laboratory fume hood.

5.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

The site health and safety plan should be reviewed with specific emphasis placed on the protection program planned for the sampling tasks. Standard safe operating practices should be followed such as minimizing contact with potential contaminants using respirators and disposable clothing as deemed necessary. At a minimum, skin protection will be afforded by disposable protective clothing. For example

Volatile organic compounds:

- Avoid breathing constituents venting from a borehole drill cuttings or trenching
- Pre-survey the borehole drill cuttings and trenches with an FID/PID prior to sampling.
  - If pre-survey results indicate parts-per billion (ppb) levels of organic constituents, sampling activities may be conducted in Level D protection.
  - If pre-survey results indicate parts-per million (ppm) levels or higher of organic constituents, sampling activities should be conducted in Level C protection.

Physical hazards associated with soil sampling are:

- Lifting injuries associated with drill moving equipment
- Pinch/crush injuries associated with drilling and hammering
- Heat/cold stress because of exposure to extreme temperatures
- Slip, trip, fall conditions because of discharge, and
- Restricted mobility due to the wearing of protective clothing.

Methanol is a toxic and flammable liquid and must be handled with all safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided and vials opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area wearing protective gloves

and safety glasses. Methanol should be stored away from open flames, areas of extreme heat, and other ignition sources.

Aqueous sodium bisulfate is a strong mineral acid. Therefore, solutions must be handled with all safety precautions related to mineral acids. Protective clothing (gloves, safety glasses, etc.) should be worn when vials containing sodium bisulfate are handled.

5.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management.

5.4 Waste Management

The EPA Region 9 Laboratory FAB Team complies with all applicable rules and regulations in the management of investigation derived waste (IDW). All team members must collect and manage IDW in a manner consistent with applicable site specific work plans. Solid and hazardous wastes are disposed of in compliance with site standards, hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

6 SAMPLE HANDLING AND PRESERVATION

Decontaminate all sampling devices and wrap in aluminum foil until needed. Each sampler should be used for only one sample. Dedicated samplers for sediment samples may be impractical when collecting numerous samples. In this case, decontaminate samplers in the field using SOP 1230 - *Sampling Equipment Decontamination.*

Samples for volatile organic analysis must be collected directly from the devise, before mixing the sample, to minimize loss due to volatilization of contaminants. Samples should be transferred to a VOA vial with a chemical preservative within 24-hours of collection. If this is done, a 14-day holding time will apply. If this action cannot be performed, either due to carbonaceous soils scheduled for low level analysis, or due to laboratory logistical issues, the samples should be stored in a freezer (-12°C). Such samples can be held for up to 7-days after sample collection.

Samples should be shipped the day of collection for overnight delivery to the laboratory. If

overnight transit is not feasible due to site logistics, samples should be held at 4□C until shipping. Samples collected in the Encore□ sampler should be received at the laboratory within 4 days of sampling. *Note: DOT regulations associated with the use of preservatives in the field may be avoided by using Encore□ samplers*

Chemically preserved samples should be stored at 4□C until analysis. A 14-day holding time is applicable. Depending on the quantity and method of packaging, sodium bisulfate and methanol may be DOT Hazardous Materials and may be subject to the DOT and International Air Transportation Association (IATA) hazardous materials regulations.

Field personnel involved in the shipment of samples prepared in the field for laboratory analysis by Method 5035, should be aware of the pertinent EPA, Department of Transportation (DOT) and International Air Transportation Association (IATA) regulations so that regulatory compliance can be maintained. Three levels of regulations apply depending on type and quantity of preservative and method of packaging. These regulations are summarized in Appendix A:

7 INTERFERENCES

Substrate particle size and organic content are directly related to water velocity and flow characteristics of a body of water. Contaminants are more likely to be concentrated in soils typified by fine particle size and a high organic content. This type of soil is most likely to be collected from depositional zones. In contrast, coarse soils with low organic content do not typically concentrate pollutants and are found in erosion zones. The selection of a sampling location can, therefore, greatly influence the analytical results.

Contamination of preservatives could result in a high bias of data. Personnel should optimize handling preservatives and sample vials in laboratory with controlled conditions. When samples are preserved in the field, it is especially critical to avoid the introduction of contamination from external sources. Consequently, personnel should work upwind of any possible source of VOCs (emissions from engines and backhoes, tobacco smoke, etc.) while adding preservatives to soil samples.

Free methanol or acidic solutions can solubilize contaminants in ambient air. Forethought while sampling or handling vials is crucial to avoid possible contamination. Do not leave vials open and exposed to ambient air. Equipment blank and field blanks should be specified in the sampling plan.

8 APPARATUS AND MATERIALS

8.1 Equipment needed for collection of soil samples includes:

- □ maps/plot plan
- □ Global Positioning Receiver/Recorder
- □ safety equipment--photoionization detector, OVM

- ☐ compass
- ☐ tape measure
- ☐ survey stakes, flags, or buoys and anchors
- ☐ camera and film
- ☐ stainless-steel, plastic, or other appropriate composition bucket
- ☐ 4-oz, 8-oz, and one-quart, wide-mouth jars w/Teflon-lined lids
- ☐ Ziploc plastic bags
- ☐ Logbook
- ☐ purge and trap grade methanol
- ☐ sample jar labels
- ☐ disposable plastic syringes
- ☐ glass vials, 40 mL, screw cap, TFE lined, septum sealed
- ☐ TFE coated magnetic stir bars
- ☐ portable top-loading balance ±0.01g
- ☐ balance weights for reference and calibration once per day
- ☐ chain-of-custody forms
- ☐ custody seals
- ☐ field data sheets
- ☐ cooler(s)
- ☐ ice
- ☐ decontamination supplies/equipment
- ☐ spade or shovel
- ☐ scoop
- ☐ bucket auger
- ☐ hand auger
- ☐ extension rods
- ☐ T-handle
- ☐ power auger
- ☐ backhoe
- ☐ drill rig

Reagents are not used for preservation of soil samples. Decontamination solutions are specified in SOP 1230 - *Sampling Equipment Decontamination.*

9 SAMPLING PROCEDURES

9.1 Preparation

Project preplanning is essential to successful sample collection. The following are prerequisites that should be addressed in advance of a full-scale field mobilization.

- ☐ Determine the extent of the sampling effort, the sampling method(s) employed, and required equipment and supplies according to the sampling QA plans for the site.
- ☐ Obtain necessary sampling and monitoring equipment.

- Decontaminate or pre-clean equipment, and ensure that it is in working order.
- Prepare schedules, and coordinate with staff, client, and regulatory agencies, if appropriate.
- Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.
- Use stakes, flags, or buoys to identify and mark all sampling locations. Specific site characteristics, including flow regime, basin morphometry, soil characteristics, and depth of overlying aqueous layer, and extent and nature of contaminant should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

Whether sampling from the surface or from depth using such devices as a split spoon, collection of the sample will be the same. Samples should be collected as quickly as possible (< 10-15 seconds). *Temporary storage of soil in split spoons, jars, or ziplock bags is not permitted.* Field screening may still be used to decide which samples will be submitted for analysis but all potential samples must be immediately chemically preserved or placed in a coring device. All protocols have been written assuming that both medium and low level sample will need to be collected.

In order to help maintain the physical structure of samples, for cohesive granular material, a hand-operated coring device must be used to collect samples of appropriate size for laboratory analysis (e.g., cylindrical soil plugs are extruded into vials using disposable plastic syringes with the tapered front ends removed). Field personnel transfer samples into pre-weighed vials containing liquid preservatives (e.g., sodium bisulfate solution or methanol). The vials are weighed in the laboratory before use and are subsequently reweighed at the laboratory after the sample aliquots are added to obtain the net sample weights.

Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed and shipped to the field location. Gloves should be worn during the preparation steps.

9.2 Low level samples field-preserved with sodium bisulfate solution

9.2.1 Add a clean magnetic stirring bar to each clean vial. If the purge and trap device employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

9.2.2 Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If sample volume is markedly smaller or larger than 5 g, adjust the amount of preservative at a ratio of 0.2 g preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of <2.

9.2.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority

of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

9.2.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted vials are used, seal both ends as recommended by the manufacturer.

9.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures the tare weight of the vial includes the label. (The weight of any added markings is negligible.)

9.2.6 Weigh the prepared vial ± 0.01 g, record the tare weight, and write it on the label.

9.2.7 As VOCs will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards must be introduced in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis

9.3 Medium level samples field preserved/extracted with methanol

9.3.1 Add 10 mL of methanol to each vial.

9.3.2 Seal the vial with the screw-cap and septum seal.

9.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures the tare weight of the vial includes the label. (The weight of any added markings is negligible.)

9.3.4 Weigh the prepared vial ± 0.01 g, record the tare weight, and write it on the label.

9.3.5 Surrogates, internal standards and matrix spikes (if applicable) must be added to the sample in the laboratory and prior to analysis.

9.4 Field collection with coring type samplers followed by laboratory preservation

Expose a fresh surface using a clean spatula or other suitable tool. Collect a sample using a coring device (e.g., the hand-held Encore□) and immediately cap following manufacturer's directions. Collect two five gram cores for the low level (if needed) and one five gram core for the medium level. Label cores and transfer to laboratory on ice as soon as possible.

9.4.1 Unconsolidated Materials

Certain soil types may not be sufficiently consolidated to collect a core sample. Two examples would be dry sand or sludges/sediments with a very high moisture content. In such cases, the plunger of the Encore□ should be pulled

back and locked. The Encore□ should be held with the opening facing upward and the sample transferred by spatula or pouring until the Encore□ is filled. The Encore□ is then capped and handled as previously outlined. *Note: Samples which are unconsolidated should be labeled as such on the chain of custody so that the laboratory can handle these samples with additional caution.*

9.4.2 Aggregate or Cemented Material

The Encore□ sampler should not be used for these materials. It can only be used for soil types that can be collected using a small diameter coring device. For other materials, the only collection technique which will maintain the integrity of the sample is field collection with methanol–protocol.

9.5 Low level soil samples field preserved with Sodium Bisulfate

The sample vials for the low-level method are designed to be placed directly in the laboratory's instrument so that they remain hermetically sealed until the VOCs are withdrawn during analysis. The entire content of each vial is processed during analysis. Hence, when low-level VOC analyses are required, it is necessary to collect at least two co-located samples. This gives the laboratory an opportunity to perform an additional analysis should the first analysis be unacceptable. Since the vials remain sealed, dilutions cannot be performed. When low-level VOC analyses are required, an extra co-located sample for the medium-level method must be collected with each set of low-level samples. Also aqueous acidic solutions are used to preserve samples for the low-level analyses therefore, low-level samples must be initially tested for carbonate interferences in the field before samples are collected.

9.5.1 Laboratory Preparation

Add 1 gram sodium bisulfate, clean magnetic stir bar, and 5 milliliters (mL) of de-ionized water to a 40 mL VOA vial. Label vial and record weight ± 0.01 grams.

Note: VOA vials with special low bleed septa must be used to prevent false positives due to siloxane peaks from standard septa. Teflon coated stir bars absorb VOCs. This is a potential loss of VOCs. Disposable stir bars should be used or if stir bars are to be re-used, the stir bars should be cleaned and the cleanliness verified.

9.5.2 Field Sampling

9.5.2.1 Samples should be collected using a coring device (modified plastic syringe) as a transfer tool. A simple coring device can be made by cutting off the front part (with tip) of a disposable non-lubricated syringe, removing the rubber plunger tip and (with repeated

experimentation) marking the length of core (2--3 cm) that corresponds to 5.0 ± 0.5 g. *Note: Use disposable syringes are NOT lubricated since so as to avoid contaminating the VOC sample.*

9.5.2.2 If the test sample passes the initial test for both effervescence and pH (section below), use the plastic syringe to collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most.

9.5.2.3 Transfer the 5 g soil sample ("or plug") into the prepared and pre-labeled sample vial by placing the syringe tip inside the vial and squeezing the syringe plunger. Cap immediately and carefully wipe the exterior of the sample collection device with a clean cloth towel.

9.5.2.4 For each sampling point, use a new plastic syringe to collect soil. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap.

9.5.2.5 An initial test sample should be collected to evaluate effervescence and chemical preservation (pH □ 2). A five gram core should be placed in a VOA vial which contains the acid solution. If effervescence occurs, the sample should be collected in a VOA vial with no sodium bisulfate. (Note: if effervescence does occur, immediately unscrew the cap to release built up pressure.) The unpreserved sample should be analyzed within 48 hours, the holding time. Results from the analysis may be biased low and should be flagged as estimated.

9.5.2.6 The test sample must also have the pH evaluated either by a pH meter or test strip to ensure that the pH has been reduced to <2 to limit biodegradation. If the sample has not been properly acidified, there are two options:

- □ Vials can be used which contain a higher amount of sodium bisulfate. This additional sodium bisulfate should be added in the lab when the vial is prepared since addition in the field would affect tare weight. The exact amount of sodium bisulfate will be determined by the buffering capacity of the soil. Sodium bisulfate can be added in the field if the field personnel record the weights of the additional preservative and sample.
- □ Analyze the sample within the 48 hour holding time and flag the data as estimated.

9.5.2.7 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 g ± 0.5 g of sample were added. The balance should be calibrated in the field using an

appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

9.5.2.8 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of soil in the syringe that corresponds to 5.0 + 0.5 g. Discard each trial sample.

9.5.2.9 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

9.5.2.10 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1- 2 grams instead of 5 grams as described above.

NOTE: When the low level samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Additional steps may be required to preserve the samples. Such steps include: addition of larger amount of the sodium bisulfate preservative to non-calcareous samples, storage of low level sample as -12□C, or significantly reduce the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel.

9.5.2.11 Record weight and transfer to ice.

9.5.2.12 A duplicate low level sample should be collected for the laboratory since low level samples cannot be reanalyzed. Ship to the laboratory per DOT regulations. (Corrosive.) *Note: Additional samples need to be collected for matrix spikes or other QC objectives.*

9.5.3 Aggregate or Cemented Material

Coring tools should not be used for these materials. For other materials, the only collection technique which will maintain the integrity of the sample is field collection with methanol–protocol below.

9.6 Medium level samples field preserved with Methanol

This particular sampling protocol has been suggested by some as a combined

preservation and extraction procedure. Carbonates are not problematic for methanol preservation and methanol sample extracts may be diluted in the laboratory when concentrations exceed the calibration range of the instrument. In addition, when samples are preserved with methanol, field personnel are not limited to single grab samples (as in the low-level method) but may composite subsamples from several locations.

9.6.1 Laboratory Preparation

Add 5 mL of methanol to a 40 mL VOA vial. Label vial and record weight ± 0.01 grams.

9.6.2 Field Sampling

9.6.2.1 Collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 g ± 0.5 g. Discard each trial sample.

9.6.2.2 Using the appropriately sized sample collection device, quickly (within 1-2 minutes exposure to the atmosphere) collect approximately 5 g of sample from the surface of the soil or sediment. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

9.6.2.3 Using the collection device, add about 5 g (2--3 cm) of soil to the vial containing 10 mL of methanol. Brush any soil off the vial threads and seal the vial with the septum and screw-cap. Total sub-sampling time should not to exceed 5 minutes. Store the samples on ice at 4□C± 2°C.

9.6.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 g ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

9.6.2.5 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate the sensitivity of the overall analytical procedure is appropriate for the intended application.

9.6.2.6 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in above. Samples collected in methanol should be shipped as described in Attachment A and must be clearly labeled as containing methanol, so the that the samples are not analyzed using the closed-system purge and trap equipment described in this procedure.

NOTE: Collection of medium concentration soil samples that are NOT preserved in the field generally follow similar procedures as for the other types of samples described above, with the exception that the

sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

9.6.3 Large Aggregate and Cemented Materials

Sample will need to be placed in a larger, wide mouth, 4 0z. glass jar and preserved with a proportionately larger volume of methanol (to maintain the 1:1 ratio). In this event, the weight or volume of methanol must be recorded.

9.7 No preservation

Under limited circumstances, Region 9 will permit the collection of unpreserved samples, such as hard or cementitious materials, debris, or large aggregates which cannot be easily collected using the options above. Field methanol preservation is the preferred approach for these types of materials. Losses of VOCs are likely and all results should be considered as estimated values.

9.8 Moisture Content Sample

In addition to the samples collected as described above, a separate container must be collected to determine moisture content. This sample can be any conveniently sized container, of glass or plastic. Ordinary soil sampling procedure are used to collect samples to measure moisture content. If samples are being collected for other analytes (e.g. metals, semivolatiles) that sample container can serve as the container for moisture content.

10 QUALITY CONTROL

The EPA Region 9 Laboratory operates a formal quality control program and tracks compliance using the Lab QC Database.

10.1 Quality Control Samples

10.1.1 Field duplicate samples should be collected at a frequency of 10 percent, or as otherwise specified in project specific work plans to assess sampling precision.

10.1.2 Equipment or rinsate blanks should be collected if equipment is field cleaned and re-used or when necessary, to document that low-level contaminants were not introduced by sampling tools.

10.1.3 Ambient or field blanks should be collected at the discretion of the field team leader based on extreme site conditions (e.g., strong odors, vapors, dust storms) or as otherwise specified in project specific work plans to document that low-level contaminants were not introduced from the airborne sources.

11 DOCUMENTATION

Sample documentation includes chain-of-custody forms and custody seals.

11.1 Chain-of-Custody

11.1.1 EPA chain-of-custody forms (Appendix B) will be used to document sample collection and transportation to the laboratory for analysis. The chain-of-custody record will identify the contents of each sample cooler and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are transported to the laboratory, the custody of the samples will be the responsibility of the sampler.

11.1.2 The sample location on the chain-of-custody form should be sufficiently descriptive so that another person could resample the same location. Other identifiers such as permanent sample station numbers, or global coordinates should also be recorded.

11.1.3 Use the “Remarks” column to record additional information and any problems encountered sampling.

11.1.4 When finished sampling be sure that all samples are recorded on the chain-of-custody form and that the sample locations and sample times on the form match those on the sample bottles. The sampler’s name, address and phone number will also be written on the form. The sampler will sign the chain-of custody record and the "relinquished by" box and note date and time when the samples are delivered to the courier service or the Regional Laboratory Sample Custodian.

11.2 Sample Collection Information.

All sample collection information pertinent to the Project will be recorded in a bound Collected Sample Logbook. Each Collected Sample Form (Appendix C) will be consecutively numbered, dated, and signed. All entries will be made in indelible ink and all corrections will consist of line-out deletions that are initialed and dated. The person making the correction will provide a brief explanation for the change. There should be no blank spaces on the form. A single line or “Not Applicable” or “NA” should be written in spaces where no information was collected. Entries may include some or all of the following information:

- Date (yyyymmdd)
- Time (military format)
- Depth start of sample
- Depth end of sample
- Units
- Sample type
- Sample method
- Analytical Method
- Sample Preservation
- Sample matrix
- QC Information:
- Rinse blank ID,
- Trip blank ID,
- Duplicate sample ID,
- Split Sample ID
- Requested
- Sample container/no. of containers
- color, texture,
- FID, PID, RAD
- Weather
- Temperature
- Comments and
- Signature of Sampler and Date

11.3 SOP Distribution and Acknowledgement

After approval, distribute an electronic copy of the final SOP to all field and laboratory staff expected to perform the SOP or review data generated by the SOP. (The Lab QC Database contains a list of assigned analysts for each SOP). All approved EPA Region 9 Laboratory SOPs are maintained in the Lab QA Sharepoint site in Adobe Acrobat portable document format.

Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Lab QC Database.

11.4 SOP Revisions

Revisions to this SOP are summarized in Appendix D.

12 REFERENCES

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APPENDIX A.
EPA, DOT, IATA REGULATIONS

Pertinent EPA, Department of Transportation (DOT) and International Air Transportation Association (IATA) regulations

- ☐ Small quantity exception--(< 30 mL inner containers), not subject to Hazardous Material Regulations (HMR) provided package is in accordance with 49 CFR 173.4.

- ☐ Limited quantity DOT hazardous material--must meet regulatory requirements minus UN specification containers (49 CFR 172.700 training applies)

- ☐ Fully regulated DOT hazardous material--Ltd Qty exception not taken, package must be in full compliance with HMRs (49 CFR 172.700 training applies)

1. Shipment as a Small Quantity Exception (49 CFR 173.4)

The DOT small quantity exception described in 49 CFR 173.4(a)(1)(i) states that the maximum quantity of material per inner container is limited to thirty (30) mL for authorized liquids, other than Division 6.1, Packing Group I materials (i.e., poisons). As applied to the preservatives of Method 5035, if there is less than or equal to 30 mL of methanol or aqueous sodium bisulfate solution per inner container (VOC vial), this material is not subject to any other requirements of the hazardous materials regulations except those presented in 49 CFR 173.4. However, aside from the 30 mL receptacle limit, there are additional restrictions:

- Each inner receptacle with a removable closure, has its closure held securely in place with wire, tape or other positive means.
- Unless equivalent cushioning and absorbent material surrounds the inside packaging, each inner receptacle is securely packed in an inside packaging with cushioning and absorbent material that (i) will not chemically react with other material and (ii) is capable of absorbing the entire contents (if liquid) of the receptacle.
- The inside packaging is securely packed in a strong outside packaging.
- The completed package, as demonstrated by prototype testing, is capable of sustaining each of the following free drops made from a height of 1.8 m (5.9 feet) directly onto a solid unyielding surface without breakage or leakage from any inner receptacle and without a substantial reduction in the effectiveness of the package:
- One drop flat on bottom
- One drop flat on top
- One drop flat on the long side
- One drop flat on the short side
- One drop on a corner at the junction of three intersecting edges
- A compressive load as specified in 49 CFR 178.606(c)

The gross mass of the completed package must not exceed 29 kg (64 pounds). The package must not be opened or otherwise altered until it is no longer in commerce. The shipper must certify conformance with this section by marking the outside of the package with the statement: This package conforms to 49 CFR 173.4," or, until 1 October 2001, with the statement: This package conforms to the conditions and limitations specified in 49 CFR 173.4. Furthermore, the shipper must indicate on the air waybill under nature and quantity of goods: Dangerous Goods in

Excepted Quantities.

IATA also requires the application of an excepted quantities label. This label contains the certification language identified above. Label entries include shipper signature, title, date, address and indication of the hazard class and associated UN number.

While 49 CFR 173.4 does not have a total net quantity limitation, IATA Dangerous Goods Regulations (DGR Section 2.7.4.2) does. For packing group II materials (e.g., methanol and sodium bisulfate), the total net quantity limit is 500 mL. This equates to 60 inner containers (VOC vials) containing approximately 8 mL of material (sample plus preservative) per outer package (i.e., sample cooler).

When discussing the shipment of DOT hazardous materials in the air mode, shippers have additional restrictions that are identified in Columns 9A/9B of the 49 CFR 172.101 hazardous materials table. Net quantity limits for methanol for passenger and cargo aircraft are one (1) liter and sixty (60) liters, respectively. The net quantity limits for sodium bisulfate solutions are one (1) liter and thirty (30) liters, respectively. Shippers should note that these quantities exceed the IATA small quantity exception. Therefore, if preservative volume (methanol or sodium bisulfate solution) is less than 30 mL per VOC vial (inner container) and the total net quantity per cooler (outer package) is limited to 500 mL, DOT HMRs or IATA DGR's quantity limits are never an issue provided packaging conforms with 49 CFR 173.4.

If more than 30 mL of methanol is used per VOC vial, shippers must address regulations for DOT-regulated hazardous material.

2. DOT Regulated Hazardous Materials Shipments, Limit Quantity

Personnel offering chemically preserved environmental samples for shipment in commerce in inner packaging (containers) containing more than 30 mL of methanol are Hazmat employees and are subject to the DOT training requirements in 49 CFR 172.700. If these individuals do not possess DOT training and do not have an employer certification, it is a violation of DOT regulations to offer these materials for transportation in commerce! Also, some generally used air shipping couriers may not ship hazardous materials or limited quantity hazardous materials. It is recommended that the proposed carrier be consulted in advance to determine if there are any company-specific requirements or limitations.

Methanol-preserved samples in greater than 49 CFR 173.4 inner-container quantities will void the 49 CFR 173.4 small quantity exception. These materials meet the definition of a DOT flammable liquid. On the shipping paper, these samples must be described using any of the following text:

- Methanol solution, 3, UN1230, PGII
- Methanol solution, 3, UN1230, PGII Limited Quantity
- Methanol solution, 3, UN1230, PGII Ltd Qty.

(Note that methyl alcohol may be substituted for Amethanol.)

It is emphasized that DOT allows for a limited quantity exception. Under the limited quantity exceptions, packages need not be UN specification. Labels are not required unless the shipment

is by air. Additionally, limited quantity shipments are not subject to placarding requirements. There are restrictions on the type of combination packaging that is acceptable for use. Since methanol is a PGII flammable liquid, 49 CFR 173.150 states the inner packaging limitation is one (1.0) liter. The outer packaging is described in the regulations as a strong outer package (i.e., a box, can, or cooler). Marking requirements must be met. An outline of the limited quantity exceptions and requirements for methanol is as follows:

Packaging:
Inner packaging: Plastic or glass < 1.0 liters
Outer Packaging: Strong outer package
Gross Weight: 66 lb (30 kg)

Labeling: Not required unless shipped by air
Primary Hazard: Flammable Liquid
Secondary Hazard: Poison

Marking: The outer package must be marked with the following items:

1. Proper shipping name: Methanol Solution
2. UN Number: Not required for Ltd Qty shipment/Otherwise required
3. DOT specification orientation arrows (See 49 CFR 172.312 for exception)
4. Shipper or receiving facility name and address
5. Cargo Aircraft only may be required depending on quantity shipped.

Shipping Paper:
1. Complete DOT shipping description
2. Number of containers (i.e., complete package)
3. Weight
4. Emergency Response information (ERG #)
5. Emergency Contact information

3. DOT Regulated Hazardous Materials Shipments, Fully Regulated

If shippers do not take a limited quantity exception and their materials are regulated in commerce, they must have DOT specification packages and would probably have to consider the cooler a DOT over pack (49 CFR 173.25). All inner packaging must be marked and labeled. Also, since the inner markings and labels will not be visible, the over pack must be marked and labeled on the outside and be marked with the following statement:

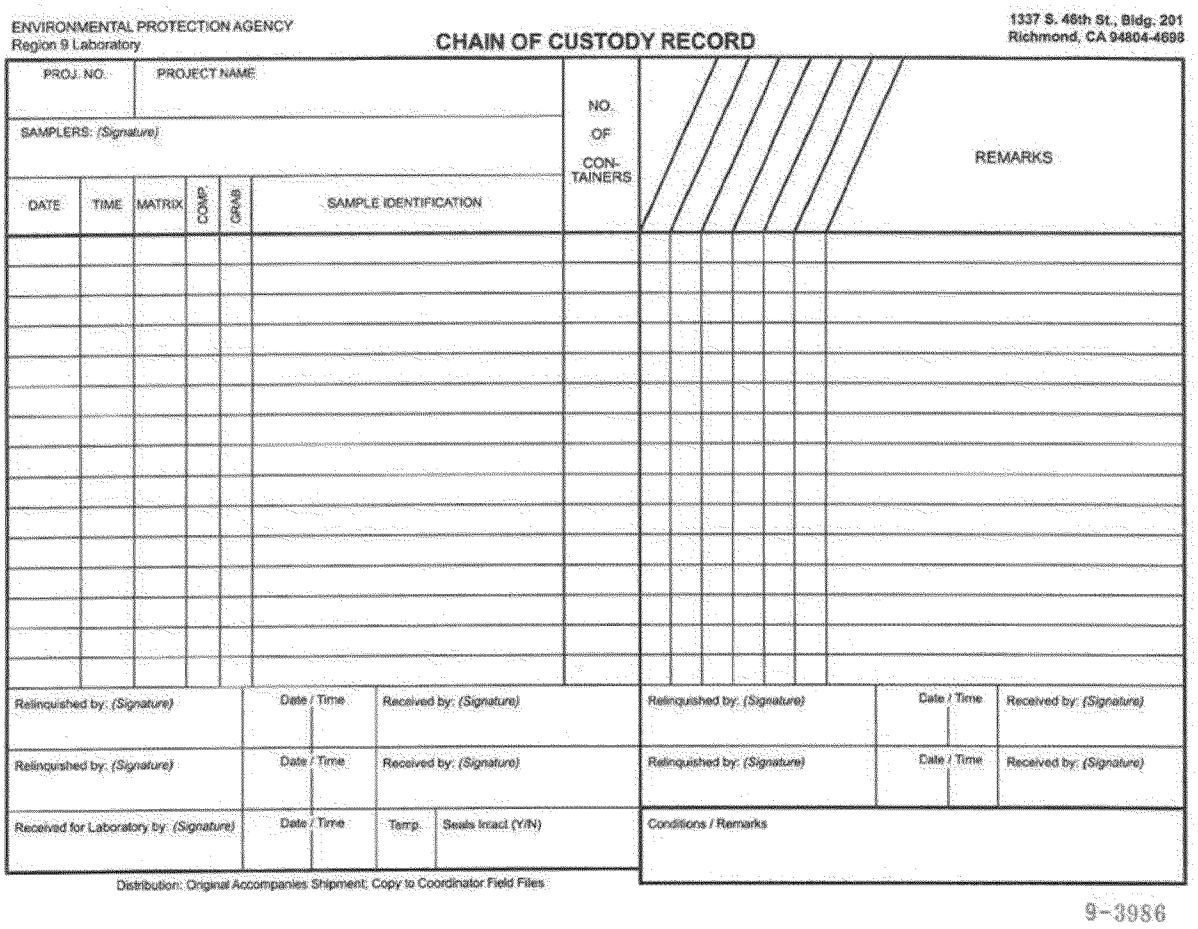
Inside (inner) packages comply with prescribed specifications

This means that the inner receptacles (glass jars or vials) must be in an authorized (UN specification) outer package. These combination packages would then be placed in the cooler (DOT over pack). For this case, all DOT shipping paper, labeling, marking (including UN numbers), and placarding requirements in 49 CFR 171 - 177 apply.

4. RCRA Regulations

1. The RCRA hazardous waste regulations are also be applicable to shipping of chemically preserved samples. 40 CFR 261.4 discusses the RCRA exemption for shipping samples. These regulations provide an exemption from the hazardous waste regulations for "samples" but not for materials which are not analyzed. Materials preserved with aqueous sodium bisulfate or methanol, which are not considered "samples," would be classified as hazardous wastes due to characteristics (corrosivity and ignitability) and would need to meet the RCRA manifesting and shipping requirements in 40 CFR 262.

APPENDIX B.
CHAIN OF CUSTODY FORM



ENVIRONMENTAL PROTECTION AGENCY
Region 9 Laboratory

CHAIN OF CUSTODY RECORD

1337 S. 46th St., Bldg. 201
Richmond, CA 94804-4698

PROJ. NO.	PROJECT NAME					NO. OF CON-TAINERS	REMARKS
SAMPLERS: *(Signature)*							
DATE	TIME	MATRIX	COMP.	GRAB	SAMPLE IDENTIFICATION		

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Received for Laboratory by: *(Signature)*	Date / Time	Temp. / Seals Intact (Y/N)	Conditions / Remarks		

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

9-3986

APPENDIX C.
COLLECTED SAMPLE FORM – SOIL/SEDIMENT

Installation/Site:_____ Area/OU:_____

Date:_____ Time:_____

Location ID:_____ Field Sample ID:_____

Begin Depth:_____ ☐ feet ☐ meters End Depth::_____ ☐ feet ☐ meters

NCoord:_____ ECoord:_____

Coord Type: ☐ UTM ☐ STP ☐ GEO Survey Type: ☐ Optical ☐ GPS ☐ Estimate

Datum: ☐ NAD83 ☐ WGS84 ☐ Other: _____ Coord Precision (±) :_____ ☐ feet ☐ meters

Elevation: _____ ☐ feet ☐ meters Elev. Type: ☐ Optical ☐ GPS ☐ Estimate

Photo Reference:_____ Elevation Precision (±) :_____

Sample Information

Sample Type	**Sample Method**	**Sample Container**
☐ Normal (N)	☐ Trowel	☐ Stainless Steel Sleeve
☐ Field Duplicate (FD)	☐ Scoop	☐ Acetate Sleeve
☐ Split Sample (SP)	☐ Hand Auger	☐ 8 oz. WM Glass Jar
☐ Trip Blank (TB)	☐ Power Auger	☐ 4 oz. WM Glass Jar
☐ Rinse Blank (RB)	☐ Cone Penetrometer	☐ Ziplock Bag (metals only)
☐ Performance Eval. (PE)	☐ Split Spoon	☐ Encore (VOCs only)
	☐ Other	☐ 40 mL vial w/ (CH_3OH)
		☐ 40 mL vial w/ ($Na_2H_2SO_3$)
		☐ Other: _____

Weather Notes:

Start: ☐ Clear ☐ Overcast ☐ Rain Temperature Start:_____ ☐ (°F) ☐ (°C)

Stop: ☐ Clear ☐ Overcast ☐ Rain Temperature Stop:_____ ☐ (°F) ☐ (°C)

Comments:_____

_____

_____

_____ _____ _____

Sampler (Print): Sampler (Sign) (Date)

APPENDIX D.
REVISION HISTORY

STANDARD OPERATING PROCEDURE: 1225
Revision: 0, Effective: 2/10/2016

SOIL SAMPLING FOR VOLATILE ORGANIC COMPOUNDS (VOCs)

Revision	Effective Date	Description
0	2/10/16	1. Initial Revision. Prepared in accordance with SOP 850 *Preparation of Standard Operating Procedures.*